SINTERING METHOD FOR W-CU COMPOSITE MATERIAL WITHOUT EXUDING OF CU

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a densification process of W-Cu composite material, and more particularly, to a sintering method for W-Cu composite material without exuding of Cu.

2. Description of the Conventional Art

Generally, a W-Cu composite material has been spotlighted as a material for high voltage electric contact or a material for micro packaging, but it is difficult for the W-Cu composite material to be used for a preparation of a composite material having a dense structure.

A densification method of the W-Cu composite material is divided into an infiltration and a sintering method. The infiltration method is for preliminarily sintering W to form a skeleton having an open pore and infiltrating liquid phase Cu into the open pore. According to the infiltration method, a complete densification can be performed by permeating Cu by a capillary force, but a composition range is limited due to a limitation of a volume fraction of the open pore. Also, an isolated pore is formed in the interior of a material. Besides, the W skeleton is collapsed when a Cu melt is infiltrated into the W skeleton at high temperature, thereby

having a difficulty in forming a uniform microstructure.

The sintering method is for mixing W with Cu metal powder and then densifying at a temperature more than a Cu melting temperature.

The densification of the W-Cu composite material according to the conventional sintering method takes place only by a particle rearrangement process due to no occurrence of solution re-precipitation process and a high contact angle between W and Cu. The particle rearrangement process means that Cu forms a liquid phase at a temperature more than a melting point 1083°C and a W particle is re-arranged. In this case, the W-Cu composite material experiences a drastic contraction and is densified. The W-Cu composite material is densified up to a relative density of approximately 90% by the particle rearrangement process, and then has a relative density of approximately 95% by a densification due to a grain growth process of the W. However, if the densification process is not completed during the particle rearrangement process and a densification by a W grain growth process is performed, a phenomenon of an exuding of Cu is taken place. The phenomenon tends to be accelerated towards a lower part of a sample. Also, the higher a Cu composition is and the larger the sample is, the phenomenon is more accelerated.

Due to the exuding of Cu, it was difficult to control a desired Cu composition in the conventional sintering method for a W-Cu composite material, and it was difficult to obtain a sample with a uniform microstructure and to control a precise dimension. Furthermore, the phenomenon that Cu exudes troubles more in case that a component shape has to be sophisticated and a composition control has to be precisely performed.

Meanwhile, it has been reported that the densification of W-Cu composite

is increased with increasing degree of missing between W and Cu and with decreasing particle size of W powder. To this end, W and Cu oxides are mechanically mixed and milled, and then reduced by a hydrogen gas, thereby facilitating a complete densification.

SUMMARY OF THE INVENTION

Therefore, an object of the present invention is to provide a sintering method for a W-Cu composite material without exuding of Cu by a liquid phase sintering method.

To achieve these and other advantages and in accordance with the purpose of the present invention, as embodied and broadly described herein, there is provided a sintering method for a W-Cu composite material without exuding of Cu comprising the steps of: holding a W-Cu composite power compact for a certain time at a Cu solid phase temperature or at a temperature just above a melting point and thus inducing a nearly complete densification; and sintering for a short time at a Cu liquidus temperature, thereby having a uniform microstructure and preventing Cu from being exuded on a surface of a product.

The foregoing and other objects, features, aspects and advantages of the present invention will become more apparent from the following detailed description of the present invention when taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

The accompanying drawings, which are included to provide a further understanding of the invention and are incorporated in and constitute a part of this specification, illustrate embodiments of the invention and together with the description serve to explain the principles of the invention.

In the drawings:

Figure 1 is a graph showing a sintering method for a W-Cu composite material according to the present invention;

Figure 2 is a graph showing a variation of a relative density according to a holding time of a W-Cu composite material having a composition of (a) W-25wt%Cu, (b)W-35wt%Cu, and (c)W-45wt%C sintered according to the present invention;

Figure 3 is a graph showing a sintering process for a W-Cu composite material in accordance with the conventional art;

Figure 4 is a graph showing a variation of a relative density according to a Cu composition of a W-Cu composite material sintered by the conventional method;

Figure 5A is an optical microscope picture of a W-Cu composite material sintered with 1 hour holding at 1200°C in accordance with the conventional art;

Figure 5B is an optical microscope picture of a W-Cu composite material sintered with 4 hour holding at 1100°C and then sintered with 0 hour holding at 1200°C according to the present invention;

Figure 6A is a drawing showing a W-45wt%Cu W-Cu composite material sintered with 1 hour holding at 1200°C in accordance with the conventional art; and

Figure 6B is a drawing showing a microstructure of a W-45wt%Cu W-Cu

composite material sintered with 4 hour holding at 1100°C and then sintered with 0 hour holding at 1200°C according to the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Reference will now be made in detail to the preferred embodiments of the present invention, examples of which are illustrated in the accompanying drawings.

The present invention relates to a sintering method for a W-Cu composite material without exuding of Cu comprising the steps of: holding a W-Cu composite power compact for a certain time at a Cu solid phase temperature or at a temperature just above a melting point and thus inducing a nearly complete densification; and sintering for a short time at a Cu liquidus temperature, thereby having a uniform microstructure and preventing Cu from being exuded on a surface of a product.

The process for holding the W-Cu composite powder compact at a solid phase temperature for a certain time is performed by an acceleration of a Cu solid phase sintering, and the reason is for preventing an exuding of Cu by achieving a densification to some degree and by finishing a particle rearrangement of W within a faster time in a state that Cu is melted as a liquid phase.

The process for holding the W-Cu composite powder compact at a temperature just above a Cu melting point for a certain time is performed by an acceleration of a W particle rearrangement, and the reason is for preventing an exuding of Cu by restraining a W grain growth by accelerating a particle rearrangement and inducing a densification at a region just above the Cu melting point where W particle are initially rearranged and a grain growth is performed to

the minimum.

The sintering method for a W-Cu composite material without an exuding of Cu according to the present invention comprises the steps of: holding a W-Cu composite material prepared by compacting a W-Cu composite powder at a reduction atmosphere for 0.5-10 hours at a Cu solid phase temperature or at a temperature just above a melting point corresponding to 800-1150°C; and cooling without a holding time by increasing a temperature into 1200-1400°C.

The reason why temperature and time are limited in the first step will be explained. At a temperature below 800°C, temperature is too low and thereby a solid phase sintering is not briskly performed. Also, at a temperature above 1150°C, a grain growth of W is performed and thereby an exuding of Cu can be taken place. When a sintering time is below 0.5 hour, a solid phase sintering and a liquid phase sintering are not sufficiently performed and a densification is not performed. Also, when a holding time exceeds 10 hours, the sintering time becomes too long thus not to have an economic efficiency.

The reason why temperature is limited as 1200-1400°C in the second step is because a particle rearrangement is briskly performed and an exuding of Cu is not taken place in the temperature range. If the temperature rises above 1400°C, the exuding of Cu is taken place.

The W-Cu composite powder prepared by a method disclosed in the Korean patent application No. 24857 in 2002 is prepared by mixing $WO_3/WO_{2.9}$ powder with CuO/Cu_2O, milling, and performing a heat treatment for reduction at a hydrogen atmosphere. The W-Cu composite powder has a round shape of a certain size that W powder surrounds Cu powder, and has a very uniform mixed shape and a very fine grain size thus to be able to obtain a relative density more

than 98% at the time of a sintering process.

A preparation method of the composite powder will be explained in more detail. First, W and Cu are weighed for a certain ratio with $WO_3/WO_{2.9}$ powder and CuO/Cu_2O material, and then uniformly mixed by using a turbular mixing or a ball milling method. Next, the mixture is held for 1min-5hours at a temperature of 200°C~400°C at a reduction atmosphere in the first step, then is held for 1min-5hours at a temperature of 500°C~700°C in the second step, and then is reduced for 1min-5hours at a temperature of 750°C~1080°C in the third step. The W-Cu composite powder prepared by said method has a structure that W surrounds Cu powder and has no intermediates generation and an impurities contamination. The W-Cu composite powder has a proper size and a round shape thus to have an excellent powder flow characteristic, a molding characteristic, and a powder injection molding characteristic.

Preferred embodiment

The preferred embodiment was performed by the sintering process show in Figure 1.

First, by an acceleration of a Cu solid phase sintering, W-Cu composite powder having three compositions of W-25wt%Cu, W-35wt%Cu, and W-45wt%C were respectively held for 1, 2, and 4 hours at 1000°C, and then the temperature was increased up to 1200°C thus to be cooled without a holding time, thereby performing a densification process. According to this, a phenomenon of Cu exuding was restrained, of which mechanism will be explained as follows. Even in case of a large volume fraction due to a high Cu composition in the W-Cu composite material, a densification can be performed to some degree only with a Cu solid phase sintering. Due to the densification in the solid phase step, a

rearrangement of W particles performed after Cu is melted into a liquid phase is completed faster, thereby restraining the exuding of Cu.

Second, by an acceleration of a W particle rearrangement, W-Cu composite powder, the W-Cu composite powder was held for 1, 2, and 4 hours like the Cu solid phase acceleration method at 1100°C which is a temperature just above a Cu melting point, and then the temperature was increased up to 1200°C thus to be cooled without a holding time, thereby performing a densification process. The process is for preventing an exuding of Cu by restraining a W grain growth by accelerating a particle rearrangement and inducing a densification at a region just above the Cu melting point where W particles are initially rearranged and a grain growth is performed to the minimum.

Figure 2 is a graph showing a variation of a relative density according to a holding time of the W-Cu composite material prepared according to the present invention. As shown in Figure 2A, in case of 25wt% where a Cu composition is relatively low, both the Cu solid phase sintering acceleration method and the W particle rearrangement acceleration method show a comparatively low relative density. According to a holding time increase at each temperature, the relative density is increased, but it is judged that a great density increase can not be anticipated even if the holding time is to be increased. However, it is observed that a relative density of the sample prepared according to the present invention is the same or higher than the sample shown in Figure 4.

As shown in Figure 2B, if the Cu composition is increased to 35wt%, a relative density is considerably increased. It is observed that a relative density held for 4 hours at 1000°C or 1100°C according to the present invention is the same or higher than that of Figure 4. In case that the Cu composition is the most

increased to 45wt%, the sample held for more than 2 hours at 1100°C and then increased to 1200°C thus to be cooled as shown in Figure 2C is almost densified. Also, in case of the sample held at 1000°C and then treated with a temperature increase thus to be cooled showed almost the same relative density as the sample held at 1200°C.

Comparison example

In order to compare with the present invention, a sintering was performed by the conventional method using a W-Cu composite powder. W-Cu composite powder having three compositions of W-25wt%Cu, W-35wt%Cu, and W-45wt%C prepared by mechanically mixing/milling W-Cu oxide and then reducing by hydrogen was used as an ingredient powder. The composite powder was molded with 100MPa pressure in a cylindrical die, and sintered for 1 hour at 1200°C under a hydrogen atmosphere according to the sintering process shown in Figure 3. As the result, as shown in Figure 4, the more the Cu composition was, the more increased the relative density was, and a densification more than approximately 96% was shown at 45wt%Cu.

However, in case of densifying by the conventional sintering method, a sufficient densification can not be obtained in a particle rearrangement step, and a final densification is performed in a pore removal step by a W grain growth. Also, a higher sintering temperature is required in order to accelerate an insufficient densification in the particle rearrangement step. The W grain growth and the high sintering temperature accelerate a phenomenon of exuding of Cu.

Figure 5A is an optical microscope picture of a W-Cu composite material of 45wt% in accordance with the conventional art, and Figure 5B is an optical microscope picture of a W-Cu composite material of 45wt% according to the

present invention. As shown, the phenomenon of exuding of Cu is restrained in the sample prepared according to the present invention than the sample prepared according to the conventional sintering method. Also, as shown in Figure 6, a microstructure of the W-Cu prepared by the present invention has a uniform and smaller W grain size than that prepared by a general sintering method, and the densification was performed like a general sintering method.

That can be also certified by a W grain size measuring result of a following table 1. From the table 1, it can be seen that W grains by a particle rearrangement acceleration method are finer than W grains by a general sintering method. This is because exuding of Cu is restrained and a densification is increased by accelerating a particle rearrangement and by decelerating a grain growth.

The table 1 shows an average W grain size and a relative density of samples sintered according to the present invention and the conventional method by using an image analysis at W-45wt%Cu.

[Table 1]

W-45wt%Cu	Feret diameter(μm)	Relative density(%)
1100°C/4h-1200°C/0h (the present invention)	0.764	97.5
1200°C/1h (the conventional method)	0.859	96.7

According to the densification method for a W-Cu composite material of the present invention, the phenomenon of Cu exuding is restrained and a W-Cu composite material having a uniform microstructure can be provided. Especially, the present invention can be applied to a preparation of W-Cu components having a minute and sophisticated shape, thereby being able to be applied to a powder

injection molding (PIM) which is being spotlighted recently. Also, the present invention can be applied to a preparation of a large component since the phenomenon of exuding of Cu is more generated at the time of preparing a relatively large component.

Also, the phenomenon of exuding of Cu can be restrained to a maximum and a densification can be performed by using a method that a proper time is held at a Cu solid phase step and then temperature is increased up to a periphery of a W rearrangement finishing temperature thus to be cooled, or by using a method that a proper time is held just above a Cu melting temperature and then temperature is increased up to a periphery of a W rearrangement finishing temperature thus to be cooled. This enables a precise composition control of the W-Cu composite material, so that the W-Cu composite material can have a constant characteristic. According to this, a surface post-processing cost is reduced and a component of a complicated shape required at a powder injection molding can be easily prepared by a near net-shape forming.

As the present invention may be embodied in several forms without departing from the spirit or essential characteristics thereof, it should also be understood that the above-described embodiments are not limited by any of the details of the foregoing description, unless otherwise specified, but rather should be construed broadly within its spirit and scope as defined in the appended claims, and therefore all changes and modifications that fall within the metes and bounds of the claims, or equivalence of such metes and bounds are therefore intended to be embraced by the appended claims.